

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:SSPTADEG1625

PASSWORD:

TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * Welcome to STN International * * * * *

NEWS	1		Web Page for STN Seminar Schedule - N. America
NEWS	2	MAY 01	New CAS web site launched
NEWS	3	MAY 08	CA/CAPLUS Indian patent publication number format defined
NEWS	4	MAY 14	RDISCLOSURE on STN Easy enhanced with new search and display fields
NEWS	5	MAY 21	BIOSIS reloaded and enhanced with archival data
NEWS	6	MAY 21	TOXCENTER enhanced with BIOSIS reload
NEWS	7	MAY 21	CA/CAPLUS enhanced with additional kind codes for German patents
NEWS	8	MAY 22	CA/CAPLUS enhanced with IPC reclassification in Japanese patents
NEWS	9	JUN 27	CA/CAPLUS enhanced with pre-1967 CAS Registry Numbers
NEWS	10	JUN 29	STN Viewer now available
NEWS	11	JUN 29	STN Express, Version 8.2, now available
NEWS	12	JUL 02	LEMBASE coverage updated
NEWS	13	JUL 02	LMEDLINE coverage updated
NEWS	14	JUL 02	SCISEARCH enhanced with complete author names
NEWS	15	JUL 02	CHEMCATS accession numbers revised
NEWS	16	JUL 02	CA/CAPLUS enhanced with utility model patents from China
NEWS	17	JUL 16	CAPLUS enhanced with French and German abstracts
NEWS	18	JUL 18	CA/CAPLUS patent coverage enhanced
NEWS	19	JUL 26	USPATFULL/USPAT2 enhanced with IPC reclassification
NEWS	20	JUL 30	USGENE now available on STN
NEWS	21	AUG 06	CAS REGISTRY enhanced with new experimental property tags
NEWS	22	AUG 06	BEILSTEIN updated with new compounds
NEWS	23	AUG 06	FSTA enhanced with new thesaurus edition
NEWS	24	AUG 13	CA/CAPLUS enhanced with additional kind codes for granted patents
NEWS	25	AUG 20	CA/CAPLUS enhanced with CAS indexing in pre-1907 records
NEWS	26	AUG 27	Full-text patent databases enhanced with predefined patent family display formats from INPADOCDB
NEWS	27	AUG 27	USPATOLD now available on STN
NEWS	28	AUG 28	CAS REGISTRY enhanced with additional experimental spectral property data
NEWS EXPRESS	29	JUNE 2007:	CURRENT WINDOWS VERSION IS V8.2, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 05 JULY 2007.
NEWS HOURS			STN Operating Hours Plus Help Desk Availability
NEWS LOGIN			Welcome Banner and News Items
NEWS IPC8			For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

All use of STN is subject to the provisions of the STN Customer agreement. Please note that this agreement limits use to scientific research. Use for software development or design or implementation

of commercial gateways or other similar uses is prohibited and may result in loss of user privileges and other penalties.

* * * * * STN Columbus * * * * *

FILE 'HOME' ENTERED AT 11:23:08 ON 28 AUG 2007

=> file caplus

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'CAPLUS' ENTERED AT 11:23:22 ON 28 AUG 2007

USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.

PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is held by the publishers listed in the PUBLISHER (PB) field (available for records published or updated in Chemical Abstracts after December 26, 1996), unless otherwise indicated in the original publications. The CA Lexicon is the copyrighted intellectual property of the American Chemical Society and is provided to assist you in searching databases on STN. Any dissemination, distribution, copying, or storing of this information, without the prior written consent of CAS, is strictly prohibited.

FILE COVERS 1907 - 28 Aug 2007 VOL 147 ISS 10

FILE LAST UPDATED: 27 Aug 2007 (20070827/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

<http://www.cas.org/infopolicy.html>

=> s olefin and distill

=> s olefin and distillation

105038 OLEFIN

103696 OLEFINS

159968 OLEFIN

(OLEFIN OR OLEFINS)

58989 DISTILLATION

422 DISTILLATIONS

59144 DISTILLATION

(DISTILLATION OR DISTILLATIONS)

178561 DISTN

1798 DISTNS

179306 DISTN

(DISTN OR DISTNS)

197143 DISTILLATION

(DISTILLATION OR DISTN)

L1 4941 OLEFIN AND DISTILLATION

=> propane and (propene or propylene) near sepatation

PROPANE IS NOT A RECOGNIZED COMMAND

The previous command name entered was not recognized by the system.

For a list of commands available to you in the current file, enter

"HELP COMMANDS" at an arrow prompt (=>).

=> s propane and (propene or propylene) near sepatation

MISSING OPERATOR ROPYLENE) NEAR

The search profile that was entered contains terms or nested terms that are not separated by a logical operator.

=> propane and (propene or propylene)
PROPANE IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> s propane and (propene or propylene)
83136 PROPANE
1129 PROPANES
83715 PROPANE
(PROPANE OR PROPANES)
75360 PROPENE
775 PROPENES
75697 PROPENE
(PROPENE OR PROPENES)
190060 PROPYLENE
305 PROPYLENES
190159 PROPYLENE
(PROPYLENE OR PROPYLENES)
L2 14686 PROPANE AND (PROPENE OR PROPYLENE)

=> s l1 and propane and (propene or propylene)
83136 PROPANE
1129 PROPANES
83715 PROPANE
(PROPANE OR PROPANES)
75360 PROPENE
775 PROPENES
75697 PROPENE
(PROPENE OR PROPENES)
190060 PROPYLENE
305 PROPYLENES
190159 PROPYLENE
(PROPYLENE OR PROPYLENES)
L3 207 L1 AND PROPANE AND (PROPENE OR PROPYLENE)

=> s l3 and (epoxidation or epoxide)
14673 EPOXIDATION
245 EPOXIDATIONS
14706 EPOXIDATION
(EPOXIDATION OR EPOXIDATIONS)
26255 EPOXIDN
570 EPOXIDNS
26343 EPOXIDN
(EPOXIDN OR EPOXIDNS)
28048 EPOXIDATION
(EPOXIDATION OR EPOXIDN)
50076 EPOXIDE
28851 EPOXIDES
65150 EPOXIDE
(EPOXIDE OR EPOXIDES)
L4 10 L3 AND (EPOXIDATION OR EPOXIDE)

=> d l4 1-10 abs ibib

L4 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB Procedure, for the continuous recycling an off-gas flow containing an unreacted olefin resulting by oxidation of olefins with hydroperoxides, includes following steps (1) compaction and cooling the off-gas flow, (2) separation the olefin from the off-gas flow by distillation, and (3) epoxidn. of the separated olefin with a hydroperoxide. The procedure is especially useful for epoxidn. of propene to propene oxide, whereby the off-gas flow contains propene and propane.

ACCESSION NUMBER: 2004:367231 CAPLUS
DOCUMENT NUMBER: 140:357189
TITLE: Procedure for the continuous recycling an unreacted olefin resulting by oxidation of olefins with hydroperoxides by compaction and pressure distillation
PATENT ASSIGNEE(S): BASF AG, Germany
SOURCE: Ger. Offen., 6 pp.
CODEN: GWXBX

DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10249378	A1	20040506	DE 2002-10249378	20021023
CA 2503449	A1	20040506	CA 2003-2503449	20031023
WO 2004037802	A1	20040506	WO 2003-EP11736	20031023
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, ME, SD, SL, SE, TE, UG, ZM, ZW, AM, AZ, BY, KG, KE, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, CA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003278127	A1	20040513	AU 2003-278127	20031023
EP 1558596	A1	20050803	EP 2003-769439	20031023
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
CN 1705652	A	20051207	CN 2003-80101850	20031023
MX 2005PA04183	A	20050608	MX 2005-PA4183	20050420
US 2006058539	A1	20060316	US 2005-532096	20050421
IN 2005CN00702	A	20070629	IN 2005-CN702	20050421
PRIORITY APPLN. INFO.: DE 2002-10249378 A 20021023				
WO 2003-EP11736 W 20031023				

L4 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB An olefin is treated with an epoxidizing agent to give a mixture of an epoxide and a carboxylic acid by-product which is separated by a double extraction procedure. The mixture is partitioned using a solvent immiscible with H₂O, which dissolves the epoxide, and an aqueous solvent to dissolve and extract the acid. Thus, a mixture containing epoxide deriva. of C14 1-alkenes 34.3, AcOH 19, and EtOAc 46.7% was passed at 1660 g/hr into the center of a 5 cm diameter York-Scheibel column with 20 stages, operating at 25". Water (3.3 parts/1 part AcOH) was introduced at the top of the column and 930 g/hr pentane at the bottom. The nonaq. phase withdrawn at the top contained 69% EtOAc and 0.6% AcOH while the aqueous phase at the bottom contained 99.4% AcOH and 11% EtOAc, which was separated by distillation. A similar process was used to sep. a mixture containing AcOH, propylene, propane, EtOAc, and propylene oxide.

ACCESSION NUMBER: 1970:3346 CAPLUS
DOCUMENT NUMBER: 72:3346
TITLE: Recovery of hydrophobic epoxide compounds
INVENTOR(S): Taylor, Wallace Edmondson; Sehnert, Merle F.
PATENT ASSIGNEE(S): Celanese Corp.
SOURCE: Fr., 7 pp.
CODEN: FRXXAK

DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1551419	---	19681227	FR	19680110
DE 1643852	---	---	DE	---
GB 1186333	---	---	GB	---
US 3541114	---	19701117	US	19670113
PRIORITY APPLN. INFO.: US 19670113				

L4 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB A continuous process for the epoxidn. of olefins (e.g., methyloxirane from propylene) with hydrogen peroxide using a product-stream predistn. step and unit is described and a process flow diagram presented.

ACCESSION NUMBER: 2001:581493 CAPLUS
DOCUMENT NUMBER: 135:137842
TITLE: Process for the epoxidation of olefins using a product-stream predistillation step and unit
INVENTOR(S): Hofen, Willi; Thiele, Georg; Moller, Alexander
PATENT ASSIGNEE(S): Degussa A.-G., Germany
SOURCE: Eur. Pat. Appl., 10 pp.
CODEN: EPXXDW

DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1122248	A1	20010808	EP 2000-102544	20000207
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
CA 2399129	A1	20010809	CA 2001-2399129	20010203
WO 2001057010	A1	20010809	WO 2001-EP1166	20010203
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KE, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, ME, SD, SL, SE, TE, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, CA, GN, GW, ML, MR, NE, SN, TD, TG				
BR 2001008063	A	20021105	BR 2001-8063	20010203
EP 1254126	A1	20021106	EP 2001-911586	20010203
EP 1254126	B1	20030702	---	---
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
AT 244231	T	20030715	AT 2001-911586	20010203
JP 2003521544	T	20030715	JP 2001-556860	20010203
ES 2202281	T3	20040401	ES 2001-1911586	20010203
ZA 2002005200	A	20030929	ZA 2002-5200	20020627
WO 2002003553	A	20020725	NO 2002-3553	20020725
US 2003114694	A1	20030619	US 2002-203184	20021004
US 6646141	B2	20031111	---	---
PRIORITY APPLN. INFO.: EP 2000-102544 A 20000207				
WO 2001-EP1166 W 20010203				

REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RECORD.

L4 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB The title compds. were prepared in a 2-step conversion process of olefins which were gaseous under normal conditions with AcOH (I). Thus, 1680 g. propylene (II) and 676 g. I (20% by weight solution in AcOH) were allowed to react per hr. in a 2-step reaction. The components were passed into a reactor 360 mm. long and 150 mm. in diameter. The conversion in the 1st step took place at 50° in 50 min. and the 2nd step at 100° in 10 min.; in both steps the pressure was 100 atmospheric. Unreacted II was recycled after condensation at 50°. The reaction product was passed to a distillation column and subjected to fractional distillation at atmospheric pressure. Propylene oxide (III), B. 33-5°, was removed at the top at 402 g./hr., 77.9% yield with respect to I supplied, and 77.1% with respect to the nonconverted II.

II. II conversion was 22.5% by weight and I conversion was 95% by weight. The use of Me₂CO resulted in 362 g. III/hr. (70.1% with respect to II and 73% with respect to I). II conversion was 21.4% by weight and the I conversion was 97% by weight. The following results were obtained with a U4A reactor: 358 g. III/hr.; III yield with respect to I, 69.5%. III yield with respect to II, 78%; I conversion, 98% by weight; II conversion, 19.8% by weight. The amount of distillation residue was 0.3 kg./kg. III formed while 13% CO₂ was formed with respect to 1 C atom of I. Corrosion gave 0.0001% by weight FeCl₃ in the reaction mixture with respect to I solution (1.1 g./m²/ day). The same experiment was carried out with 0.01% by weight NaSP3010 and 0.01% by weight quinoline as stabilizers. The results were: 465 g. III/hr.; III yield with respect to I, 90.1%; with respect to II, 87.6%; I conversion 97% by weight; and II conversion 22.6% by weight. Isobutylene (3420 g.) and 20% by weight I solution in EtOAc (4940 g.) was stabilized with 0.01% by weight NaSP3010 and 0.01% by weight quinoline. The mol. ratio isobutylene-I was 4.7:1; in the 1st step, the temperature was 30° and the time 30 min.; in the second step, temperature 70°, time 6 min. Pure isobutylene oxide was obtained (88.2 % with respect to I supplied).

ACCESSION NUMBER: 1966:473342 CAPLUS
DOCUMENT NUMBER: 65:73342
ORIGINAL REFERENCE NO.: 65:13655h, 13656a-c
TITLE: Alkane epoxides
INVENTOR(S): Knapsack, A.-G.
SOURCE: 11 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NL 6513640	---	19660425	NL 1965-13640	19651021
BE 671268	---	---	---	---
PRIORITY APPLN. INFO.: DE 19641022				

AB of. CA 57, 13716g. The (tert-Bu)2O2 (I)-induced free radical addition of propylene oxide (II), styrene oxide (III), 1,2-epoxybutane (IV), 1,2-epoxycyclohexane (V), and 1,2-epoxy-3-phenoxypentane (VI) to 1-octene (VII)

was studied in the temperature range 125-200°. A mixture of isomeric epoxycyclohexanes distilled gave 2,3-epoxycyclohexane, b. 56-7°, n_D20 1.3815, and IV, b. 58.5-9.0°, n_D20 1.3832. The base-catalyzed reaction of H₂O with mesityl oxide according to Payne (CA 52, 16357f) gave 4-methyl-3,4-epoxy-2-pentanone (VIII), b_D20 61-2°, n_D20 1.4235. V (2 moles), 0.40 mole VII, and 0.06 mole I deaerated with N and heated 3 hrs. at 145°, the mixture freed from peroxide decomposition products and unreacted V and VII by distillation, the residue distilled at 0.10 mm. to remove the 1:1 adduct, and the product redistd. gave 7.7% 7-hexadecanone, b_D10 82°, n_D20 1.4432, λ 5.85 μ failing to give pos. Ketone test with Brady reagent and HONH₂.HCl, or to yield a semicarbazone.

Gas-chromatographic comparison of its retention time with that of an authentic synthetic sample proved the assigned structure. IV (2 moles), 0.40 mole VII, and 0.06 mole I heated 3 hrs. at 145° in a deaerated Parr autoclave yielded 3.43 g. 3-dodecanone, b_D15 65°, n_D20 1.4425, λ 5.85, 3.0 . III (2 moles), 0.20 mole VII, and 0.03 mole I heated 3 hrs. at 150° under N and the mixture distilled gave 3.10 g. capriphenone (95% pure), b_D50 120-8°, n_D20 1.4975, 5.85 . VI (1 mole), 0.10 mole VII, and 0.03 mole I heated 5 hrs. at 150° under N, the mixture distilled, and the solid (4.45 g., b_D10 170-200°) recrystd. 4 times from petr. ether gave a product, m. 83°, λ 5.85μ, mol. weight 527, consisting of 2:1 and 3:1 addition products (mol. weight 486 and 598). These results indicated that the chain transfer

atom was the α-tertiary H atom on the epoxide ring. Formation of this product also indicated that the intermediate epoxy radicals, initially formed, rearranged to α-oxo radicals before addition to the olefin. A mechanistic path for this general reaction was outlined and discussed. II (3 moles), 0.20 mole VII, and 0.015 mole I in a deaerated steel bomb heated 17 hrs. at 125° and the mixture distilled gave 2.33 g. 5-hydroxy-2-hexanone (IX), 80% pure, b_D10 60-90°, n_D20 1.4520, mol. weight 120.7, λ 3.0, 5.85 μ,

together with 2.33 g. residue, mol. weight 640. II (2 moles), 0.20 mole VII, and 0.03 mole I heated 2 hrs. at 150° in a deaerated Parr bomb and the mixture distilled left a residue of 11.2 g., n_D20 1.4595, mol. weight 324, and

gave 5.34 g. product, b_D26 62-70°, n_D20 1.4368, mol. weight 211, λ 3.0, 5.85 μ, chromatographed to show the presence of 4.27 g. IX and 1.07 g. 2-undecanone (X). II (2 moles), 0.10 mole VII, and 0.12 mole I heated 1 hr. at 200° in a deaerated Parr bomb and the mixture distilled left 10.2 g. residue, n_D20 1.4622, mol. weight 349, and gave 4.80 g.

product, b_D26 50-7°, n_D20 1.4370, mol. weight 175.6, λ 3.0, 5.85 μ, containing 0.96 g. IX and 3.84 g. X. X semicarbazone m. 118-20°. With II, a secondary reaction, involving opening of the epoxide ring by intermediate acetyl radicals, produced IX. To ascertain the effect of ring size on the rate of H atom abstraction, the reactivities of the above epoxides II, III, IV, V, VI, VII as, well as trimethylene oxide, tetrahydrofuran, and tetrahydropyran toward Me₃CO₂ radicals were determined relative to C₆H₆. The reactant and I in

50:1 molar ratio was weighed into a glass ampul cooled in a solid CO₂-CHCl₃-CCl₄ bath, flushed with argon, the sealed ampul heated 40 hrs.

AB Olefins with more than 3 carbon atoms can be epoxidized in liquid phase by molecular oxygen using as catalyst olefinic aldehydes, that give the corresponding olefinic acids, and sulfur or aromatic nitro derivatives. Thus: A solution of 168 g. acrolein, 420 g. propylene, 180 g. nitrobenzene, and 1170 g. EtOAc is poured into a pressure vessel,

heated at 100°, and saturated with oxygen for 1 hr. The distillation of the solution gives 397.2 g. unreacted propylene, 69.3 g. acrylic acid, (64%) 84 g. unreacted acrolein, and 28.4 g. 1,2-epoxypropene (90%). A solution of 112 g. acrolein, 420 g.

propylene, 1369 g. EtOAc, and 5 g. sulfur is heated at 80°, and treated with molecular oxygen 2 hrs. to give (with recyclozation) 82% 1,2-epoxypropene, and 70% acrylic acid; without sulfur the yields are 15% and 70%, respectively. A solution of 210 g. methacrolein, 420 g. propylene, 180 g. nitrobenzene, and 1130 g. EtOAc is heated at 95° and treated with oxygen for 16 min. to give (with recyclozation) 85% 1,2-epoxypropene, and 86% methacrylic acid, (conversions 7.6% and 28%, respectively). A solution of 210 g. crotonaldehyde, 420 g. propylene, 180 g. nitrobenzene, and 1130 g. EtOAc is heated at 90° and treated with oxygen for 25 min. to give (with recyclozation) 91% 1,2-epoxypropene, and 65% crotonic acid (conversions 5.8%, and 66%, respectively).

ACCESSION NUMBER: 1965:471400 CAPLUS

DOCUMENT NUMBER: 63:71400

ORIGINAL REFERENCE NO.: 63:13083f-h,13084a

TITLE: Epoxidation of olefins in liquid

phase with molecular oxygen

INVENTOR(S): Lanos, Francois; Clement, Genevieve

PATENT ASSIGNEE(S): Institut Francais du Pétrole, des Carburants et

Lubrifiants

SOURCE: 12 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1401176		19650521	FR	19630803
PRIORITY APPLN. INFO.:			FR	19630803

at 135 ± 1° (stirred oil bath), the cooled ampul opened, and the mikt. analyzed by gas chromatography. The reactivity studies were tabulated and results indicated that 5- and 6-membered cyclic ethers underwent H atom abstraction most readily. Trimethylene oxide, II, and IV were similar to C₆H₆ in reactivity. V and VI were intermediate in reactivity, whereas III, which contains a benzylic H, was the most reactive epoxide.

ACCESSION NUMBER: 1963:421224 CAPLUS

DOCUMENT NUMBER: 59:21224

ORIGINAL REFERENCE NO.: 59:3748a,3749a-f

TITLE: Free radical chemistry of cyclic ethers. IV. Free

radical rearrangement of epoxides

AUTHOR(S): Wallace, T. J.; Gritter, R. J.

CORPORATE SOURCE: Univ. of Connecticut, Storrs

SOURCE: Tetrahedron (1963), 19, 657-65

CODEN: TETRAH; ISSN: 0040-4020

Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 59:21224

L4 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB The autoxidn. of allyl chloride (II), methallyl chloride (III), 1,4-dichlorobutene-2 (III), and 3,4-dichlorobutene-1 (IV) formed chlorohydrins by attack at the double bonds. Analysis of the initial products from II indicated an epoxide precursor was involved. Oxidation rates for III and IV were studied and show an unusual dependency on added anions. All allylic chlorides were fractionated. Oxidns. at atmospheric pressure were conducted using cylinder O₂. For pressure oxidns. an autoclave was used. Products from I and II were analyzed by gas chromatography. Without a catalyst the oxidation of II at 60° was very slow. A maximum oxidation rate of 5.5 + 10-7 mole O/mole olefin/sec. was reached in 12 hrs.; after 44.7 hrs. 0.064 mole of O/mole II was consumed, and the reaction terminated. In order to obtain a higher conversion and allow isolation of products 90.6 g. II containing 1.8 g. cobaltic acetyl-acetonate and 5 g. cumene hydroperoxide oxidized 46.3 hrs. at 60° and distillation of an 89-g. aliquot of the liquid product gave 40.7 g., b200 35-7°, 5.9 g., b200 78-9° (contained 1.6 milliequiv. of epoxide), 2.5 g., b50 55-75°, 4.5 g. b10 66-90°. At 200 lb./sq. in. 487.1 g. II containing 20 g. MgO and 4.8 g. tert-butyl hydroperoxide oxidized at 100° with an off gas, the reaction terminated at 375 min. after the consumption of 3.55 moles of O, the solid product separated, dissolved in H₂O, washed, acidified, and extracted with Et₂O gave 9.8 g. of liquid consisting mainly of HCO₂H. The 510 g. of liquid oxidation product gave a min. of 15 separate peaks on gas chromatography. Chloroacetone and 1,3-dichloro-2-methylpropanol-2 (V) constituted 24 and 11%, resp. Chloroacetone and 1,2-epoxy-3-chloro-2-methylpropane (VI) were identified by infrared gas spectra. V was purified by redistn., b14 42.8°, n_D20 1.4700, d₂₀ 1.250. The bis-2-naphthyl ether derivative m. 146-8°. Reaction with aqueous suspension of Ca(OH)₂ gave VI. Acidification of the salts yielded nearly all HCO₂H, a trace of AcOH, and no chloroacetic acid (43.2 min.). Attempts to titrate samples from various reaction stages for hydroperoxide iodometrically gave poor results. I (497.3 g.) containing 20 g. MgO at 100° and 200 lbs./sq. in. for 440 min. required 76.8 g. O and a constant maximum rate was reached in 260 min. ClCH₂CHO was identified. HCO₂H was present in the distillate. 1,3-Dichloropropanol was isolated as a pure liquid; phenylurethan m. 172-3°. The acid components (8.5 g.) extracted from the acidified MgO contained equal ams. of chloroacetic acid and HCO₂H. The oxidation of III at 90° was terminated after the consumption of 0.74 mole O at 27 hrs. Several tenths of a gram of (CO₂H)₂ was directly filtered. The solution was found to contain 2.2 milliequiv./g. of acid. The solution, 81 g., taken up in CHCl₃, washed, dried, and distilled gave 13 g. unchanged III. No 1,3-isomer was obtained. Trichlorobutanol in 28% yield was collected at 68-77°/1 mm., n_D20 1.4980. The oxidation of 2M III in AcOH with 0.1M cobaltous chloride gave 0.19 mole HCl and 0.13

L4 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 50, 24121. Me₂CHC(OH)Ph₂ (II) gave 60% Me₂CCPh₂ (III) with Ac₂O and 50% with o-C₆H₄(CO)₂ (IIa); it was best prepared by keeping 40 g. I, 50 g. C₅H₅N, and 42 g. POCl₃ at room temperature several days, pouring on ice, extracting with Et₂O, drying the ext., concentrating, distilling, refluxing the distillate 6 hrs. with Na, and redist. to give pure II, b1.6 114°, n_D20 1.586. II (20 g.) in 100 cc. Ac₂O treated in 0.5 hr., with cooling, with 15 g. Cr₂O₃ in 80 cc. Ac₂O, stirred 3 hrs., poured into 1.5 l. H₂O, and the product extracted with Et₂O gave 14.4 g. CMe₂CPH₂O (III). b0.7 102-12°, m. 64° (alc.); by-products of the reaction were identified as Ph₂CO and Me₂CO. II and Bz₂O₂ also gave III, b0.25 88°. III (0.2 g.) and 5 cc. 43% H₂SO₄ shaken several days gave a diol, m. 94-5° (petr. ether). II reacted vigorously with Cr₂O₃ in 65% H₂SO₄, giving no III, but instead, Ph₂CO and Me₂CO. p-MeC₆H₄CH(OH)CHMe₂ and saturated Et₂O-HCl gave p-MeC₆H₄CHClCHMe₂ which, treated in situ, with C₅H₅N gave p-MeC₆H₄CHMe₂ (IV), m. 58-9° (alc.). As above, 20 g. IV and 11 g. Cr₂O₃ in Ac₂O gave 13 g. epoxide (V), b1.5-1.7 139-42° [by-products were (p-MeC₆H₄)₂CO and Me₂CO]; IV and Bz₂O₂ also gave V, m. 58-9°. V, as above gave a diol, identified by oxidation with HIO₄ to (p-MeC₆H₄)₂CO (VI). IV and Cr₂O₃ in 43% H₂SO₄ gave VI but no V. p-Brc₆H₄MeBr (from 500 g. p-Brc₆H₄Br and 60 g. Mg) and 110 g. Me₂CHCO₂Et gave crude (p-Brc₆H₄)₂C(OH)CHMe₂ (VII); distillation of VII gave (p-Brc₆H₄)₂C(OH)CHMe₂ (VIII), b0.7 174°, m. 97° (alc.); as above, 15 g. VIII gave 10 g. epoxide (IX), b1.2 177-8°, m. 94-5° (alc.). IX was unaffected by 43% H₂SO₄ at room temperature for 3 days. Similarly were prepared (p-ClC₆H₄)₂C(OH)CHMe₂, (p-ClC₆H₄)₂CMe₂, b0.2 132°, m. 71-2°, and the epoxide (X), b0.65 148-50°, m. 57-8°. Me₂CHCHMeC(OH)Ph₂, b2 198-200°, n_D20 1.564, (205 g.) and 600 g. IIa gave Me₂CHCHMeCPh₂ (XI), b15 168-70°, n_D20 1.5740 and X gave the epoxide (XII), b12 172-4°, m. 93° (alc.). XII also resisted hydrolysis with 43% H₂SO₄. Me(iso-Pr)C(CO₂Et)₂ gave Me₂CHCHMeCO₂H, b. 142-6°, n_D20 1.4023.
ACCESSION NUMBER: 1958:25393 CAPLUS
DOCUMENT NUMBER: 52:25393
ORIGINAL REFERENCE NO.: 52:4561g-1,4562a-b
TITLE: Reactions of unsaturated compounds. XIII. Oxidation of 1,1-diarylolefins by chromic oxide
AUTHOR(S): Hickinbottom, W. J.; Moussa, G. E. M.
CORPORATE SOURCE: Univ. London
SOURCE: Journal of the Chemical Society (1957) 4195-8
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L4 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN (Continued)
mole CO₂/mole of III. Distn. gave no sepn. of products. An 82% yield of a liquid b3 73-160° consisted of esters contaminated with acids, olefinic, and hydroxy comds. Titration of the crude oxidn. mixt. for hydroperoxide was unsuccessful. Oxidn. of IV at 90° with 0.5% Co naphthenate consumed 0.16 mole O in 24 hrs., and purification of the product gave 35 g. IV and 7.6 g. III. No isomer was recovered for reaction in the absence of catalyst. The combined products from oxidn. of 2M IV in AcOH representing 225 g. starting material were distd. The AcOH forerun of 29 g. yielded an unstable 2,4-dinitrophenylhydrazone, m. 125°. From the 18.5 g. fraction, b4 54-66°, 4.5 g. crystals were obtained, m. 62-3°, probably ClCH₂CO₂H. The p-bromophenacyl ester m. 97-9°. The ester fraction, b4 72-81°, 14.5 g., on hydrolysis with 1% H₂SO₄ and steam distn. gave AcOH and a liquid from which 1,2,3,4-tetra-chlorobutane was obtained as platelets, m. 73°. The remaining liquid was trichlorobutanol; urethan m. 132-3°. The next highest boiling distillate, b1 81-127°, 8.6 g., appeared to be a mixt. of esters. And the last fraction of 17 g., b1.1 127° pptd. some crystals, m. 76-7°. The pot residue was 40.8 g. Pure 1,3,4-trichlorobutanol-2 (VII) was obtained by refluxing either the impure chlorohydrin or the ester fraction from the oxidn. of III or IV with 5 times its wt. of N MeOH-HCl. VII b1 80°, n_D20 1.5022.0 With base VII gave an epoxide, b44 96-100°, n_D20 1.4749; phenylurethan m. 132-3°. Reaction of the chlorohydrin with 20% NaOH gave 80% dichloroepoxybutane, b10 73-5°, n_D20 1.4767. The rate of basic hydrolysis of 0.1N chlorohydrin was second order rate. A plot of O consumed against time generally produced typical sigma shaped curves. The following results were obtained (isomer, solvent, temp., concn. of the dichlorobutene, concn. of catalyst, added salt or acid, and the rate + 104 given): IV, AcOH, 90°: 2M, 0.1M, CoOAc, 10°: 1M, 0.1M, CoOAc, 7.6°: 2M, 0.05M, CoOAc, 7.8°: 2M, 0.05M CoOAc, 0.015M HCl, 5.5°: 2M, 0.05M, CoOAc, 0.2M, LiCl, 3.2°: 2M, 0.05M, CoCl₂, 11°: 3,4-Dichlorobutene, no solvent, 90°, no catalyst, 1.0, 0.5 mole-% Co as Co naphthenate, 3.2°: 15 mole-% K₂CO₃, no reaction. CoOAc, and CoCl₂ represent cobaltous acetate-4H₂O and CoCl₂·6H₂O, resp. III, AcOH, 90°: 2M, 0.1M, CoOAc, 13°: 2M 0.1M, CoOAc, 0.4M, Pb(OAc)₂, 26°: 2M, -, CoCl₂, 30.100°: 2M, 0.1M, CoCl₂, 23.80°: 2M, -, CoCl₂, 21°: 2M, 0.1M CoCl₂, 1% 2-azobisisobutyronitrile, 20.70°: 2M, 0.1M, CoCl₂, 8°. III, no solvent, 90°: no catalyst, 10°: 0.5 mole-% Co as Co naphthenate, 6.8°, no catalyst, 1% 2-azobisisobutyronitrile, 10.80°: 0.5% Co as Co naphthenate, 20%, light MgO, 11°: no catalyst, 20%, light MgO 13°. III, Me₂CO, 90°: 0.4M, negligible reaction.
ACCESSION NUMBER: 1962:1895 CAPLUS
DOCUMENT NUMBER: 56:1895
ORIGINAL REFERENCE NO.: 56:300g-1,301a-1,302a
TITLE: Autoxidation of liquid allylic chlorides
AUTHOR(S): Brill, Wm. F.
CORPORATE SOURCE: Food Machinery and Chem. Corp., Princeton, NJ
SOURCE: Journal of Organic Chemistry (1961), 26, 2969-72
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L4 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN
AB Epoxides are prepared by treating an ethylenically unsatd. compound with AcH- or EtCHO-monoperacetyl, preferably at 40-120°. Thus, 351 g. of a mixture of AcH (75%) and Me₂CO (25%) charged to a cylindrical oxidizer, cooled to -4°, irradiated with ultraviolet light, and O forced through by means of a diffuser given 46% AcH-monoperacetyl (I) at the end of 2 hrs. Fresh AcHMe₂CO may be added at the top of the oxidizer and the oxidized product removed from the bottom. Styrene (413 g.) containing 0.8 g. 4,2,6-Cl(O₂N)₂C₆H₂OH (II) is charged to a still, refluxed at 70° under 60 mm. pressure, 229 g. of a 52% solution of I (prepared as above) added over a period of 80 min. and 10N II in styrene is added simultaneously through the reflux column at a rate of 20 ml./78 min. and the AcOH, Me₂CO, and AcH are removed continuously. Distillation of the residual material gives 86 g. styrene oxide, as well as unreacted styrene. Similarly were prepared the following from the corresponding olefins: 4-vinylcyclohexene monoxide, b9 57°; propylene oxide; 3,4-epoxy-4-methyl-2-pentanone, b26 65°; 9,10-epoxystearic acid, m. 52°; glycidol, b5 57°; chloroisobutylene oxide, b50 46°; butadiene monoxide; 3,4-epoxycyclohexanecarbonitrile, b2.5 76-85°; 9,10-epoxystearamide, N-(n-butyl)-4,5-epoxycyclohexane-1,2-dicarboximide.
ACCESSION NUMBER: 1956:44861 CAPLUS
DOCUMENT NUMBER: 50:44861
ORIGINAL REFERENCE NO.: 50:8730b-e
TITLE: Epoxidation of ethylenically unsaturated organic compounds
PATENT ASSIGNEE(S): Union Carbide & Carbon Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 735974		19550831	GB	

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN (Continued)
 AB The object of this work was the preparation and study of the phys. properties of certain hydrocarbons with b. ps. in the range of gas oil (petroleum) fraction b. 200-320°. Preparation of hydrocarbons: Aliphatic series: (1) Pentadecane (I). Condensation of (CH₂O)₃ with C₆H₁₃MgBr gave 38% heptanol; condensation of C₇H₁₅MgBr with HCO₂Et gave 61% n-pentadecanol (II), m. 43.5° (from EtOH-H₂O), b.p. 153° (cf. C.A. 41, 5850e for data on C₆H₁₃Br and C₇H₁₅Br). Dehydration of II in H₂SO₄ gave 7-pentadecene, b.p. 137°, d₂₀ 0.7726, n_D 1.4370, MR (mol. refraction) 71.21, oxidation of which with CrO₃-HOAc yielded heptanoic acid, b. 218-24°, and octanoic acid, b. 233-8°, both characterized by titration and the Ag salts. Catalytic hydrogenation with Pt black of II gave I, b.p. 145°, d₂₀ 0.7638, n_D 1.4286, MR 71.49. (2) 4,6-Dipropylnonane could not be prepared. The 4-heptanol (III) prepared in 88% yield from C₃H₇MgBr and HCO₂Et b.p. 70°, d₂₀ 0.8127, n_D 1.4184, MR 36.00; these data compared favorably with those for III prepared from C₃H₇MgBr and C₃H₇CHO, in contrast to the poorer data for III prepared from esters: b.p. 64°, d₂₀ 0.8139, n_D 1.4181, MR 35.92. The C₇H₁₅MgBr was formed with difficulty, EtBr being necessary to prime the reaction, and subsequent condensation with HCO₂Et gave only secondary products, notably 2-propyl-1-pentanol. The preparation of 3-propyl-2-(2-propylbutyl)hexanoic acid (IV) by malonic ester synthesis (for subsequent decarboxylation to C₁₅H₃₂) was unsuccessful. (3) 2,4,8,10-Tetramethylundecane could not be prepared from Me₂CHCH₂CHMeCH₂MgBr and HCO₂Et; only secondary products were obtained. 4-Methyl-2-pentanol, 81% from Me₂CHCH₂MgBr and AcH, b.p. 47°, d₂₀ 0.80708, n_D 1.4101, MR 31.22; formation of its bromide with P and Br or with HBr was difficult, and subsequent condensation with (CH₂O)₃ gave mostly secondary products and 2,4-dimethylpentanol. (4) 2,6-Dimethyl-3,5-diisopropylheptane was not formed from (Me₂CH)₂CHMgBr and HCO₂Et, but instead (Me₂CH)₂CH₂Me, Me₂CHCH₂CHMe₂, and (Me₂CH)₂CHCH₂CHMe₂ were obtained. The malonic ester synthesis was considered improbable after attempted preparation of IV. (5) Hexadecane (V), 60% from 1-bromooctane and Na, b.p. 153°, d₂₀ 0.7770, n_D 1.4351, MR 75.91; in Et₂O the reaction is slow, with an intermediate blue color; if 1-bromooctane is added to a suspension of Na in xylene, the initially fast solution of the Na is followed by the slow elimination of Br as MeBr (refluxing 3 h.) (70% yield). (6) 5-Methyl-4,6-dipropylundecane, the intermediate (P₂CH₂CH₂CHMe) could not be obtained by Grignard condensation, only secondary products such as 3-propyl-2-hexanol. (7) 4,7-Dipropyldecane (VI). 4-Heptanone (VII), from 4-heptanol and CrO₃-HOAc, b.p. 142°, d₂₀ 0.8116, n_D 1.4065, MR 34.53; semicarbazone m. 135°. BrMgC₂Cl₂ and EtMgBr (VIII) was made by passing a stream of pure C₂H₂ into Et₂O and EtMgBr until C₂H₆ evolution ceased and 2 liquid layers appeared; condensation with VII gave 89% acetylenic glycol, m. 120° (from CCl₄), dehydration of which in boiling 25% H₂SO₄ gave 4,7-dipropyl-3,7-decadiene-5-yne (IX), b.p. 142°, d₂₀ 0.8111, n_D 1.4862, MR 77.19 (an extraordinary exaltation of 4.04 was noted). Hydrogenation with Pt black gave VI with phys. data different from those found in the literature (cf. C.A. 8, 1579), b.p. 141°, d₂₀ 0.7802, n_D 1.4350, MR 75.59. Selective hydrogenation with Raney Ni in EtOH gave instead intermediate products, identified by oxidation: 4,7-dipropyl-3,5,7-decatriene, 4,7-dipropyl-4,6-decadiene, and 4,7-dipropyl-5-decene. (8) 2,4,6,8,10-

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN (Continued)
 1,2-diphenyl-2-propanol, m. 72° (from EtOH), b.p. 163°, dehydrated in boiling 50% H₂SO₄ to 1,2-diphenyl-2-propene, m. 81° (from EtOH), b.p. 170°, slightly sol. in Et₂O. Hydrogenation with Pt black was selective, to form XXI, b.p. 160°, d₂₀ 0.9799, n_D 1.5573, MR 64.42. (4) 1,3-Diphenylpropane (XXII). PhCH₂MgCl and HCO₂Et gave only 7-8% 1,3-diphenyl-2-propanol (XXIII), owing to the formation of secondary products such as PhCH₂CH₂OH; the Grignard condensation of PhCH₂CH₂Br with BzH gave 3% 1,3-diphenyl-1-propanol (XXIV), m. 71° (from EtOH), b.p. 186°. Dehydration of XXIII with H₂SO₄ gave trans-1,3-diphenylpropane, m. 51° (from EtOH), b.p. 159°; similarly XXIV gave a mixt., b.p. 170°, d₂₀ 0.9978, n_D 1.5689, MR 63.68; chilling removed 30% as the solid trans form, and the oily liq. was purified to cis-1,4-diphenylpropene, b.p. 178°, d₂₀ 1.0138, n_D 1.5807, MR 63.75. XXII, formed by hydrogenation with Raney Ni of either isomer, b.p. 160°, d₂₀ 0.9831, n_D 1.5594, MR 64.41. (5) 1,2-Diphenylbutane (XXV), b.p. 172°, d₂₀ 0.9777, n_D 1.5554, MR 68.98. Friedel-Crafts condensation of EtCOCl and C₆H₆ gave 88% Et Ph ketone, b.p. 97°, d₂₀ 1.0103, n_D 1.5256, MR 40.69, which with PhCH₂MgCl gave 1,2-diphenyl-2-butanol, b.p. 180°, d₂₀ 1.0367, n_D 1.5752, MR 72.06. Dehydration in H₂SO₄ or P₂O₅ gave a mixt. of isomeric olefins; subsequent hydrogenation with Raney Ni was selective, to form XXV, attacking only isomers without a double bond between the 2 rings (the formation of XXVIII from stilbene is an exception to this general rule). (6) 1,4-Diphenylbutane (XXVI). The 2 diastereoisomers of 1,4-diphenyl-2-butene-1,4-diol, from BzH and VIII, were sepd. by their different solubilities in Et₂O; on crystn. from EtOH the α-form m. 146° and the β-form m. 99.5°. The products, predominantly α-, resinify easily in acid. Hydrogenation gave the stereoisomeric diols: α-form (XXVII) of 1,4-diphenyl-1,4-butanediol m. 110°, β-form m. 93°. In contrast to the aliph. diols, dehydration of XXVII with 30% H₂SO₄ did not give a diolefin, but instead 1,4-diphenyl-1,4-epoxybutane (2,5-diphenyltetrahydrofuran) (XXVIII), b.p. 210°, d₂₀ 1.0752, n_D 1.5770, MR 63.49. Very stable, resistant to H in EtOH with Raney Ni or Pt black, reacts slowly with H and Pt black in HOAc. In contrast, the action of nascent H, from Na and EtOH, is very effective, giving 1,4-diphenyl-1-butanol, b.p. 218°, d₂₀ 1.0347, n_D 1.5553, MR 65.17; dehydration in 60% H₂SO₄ then gave 1,4-diphenyl-1-butene, b.p. 170°, d₂₀ 1.0330, n_D 1.5945, MR 68.37. The XXVI produced by hydrogenation with Raney Ni, m. 52.5°, was identical with that prep. from PhCH₂CH₂Br and Na. No rearrangement occurred in the dehydration of XXVII (cf. Tiffeneau, Orskoff, and Levy, C.A. 26, 2429). (7) 1,2-Diphenylpentane (XXIX), b.p. 184°, d₂₀ 0.9649, n_D 1.5480, MR 73.74. 1,2-Diphenyl-2-pentanol, 94% from PhCH₂MgCl and Pr₂Se, b.p. 184°, d₂₀ 0.9913, n_D 1.5882, MR 81.50, was dehydrated with H₂SO₄ to a mixt. (XXX) of isomeric olefins, b.p. 185°, d₂₀ 0.9868, n_D 1.5798, MR 74.85; dehydration with P₂O₅ gave a mixt. (XXXI), b.p. 196°, d₂₀ 1.0026, n_D 1.5807, MR 73.76. XXXI contains mostly 1,2-diphenyl-2-pentene and is smoothly hydrogenated at normal pressure with Raney Ni to XXIX, whereas XXX, contg. mostly 1,2-diphenyl-1-pentene, is not hydrogenated. (8) 2,5-Diphenylhexane (XXXII). 2,5-Diphenyl-3-hexyne-2,5-diol, from VIII and MeBr, was sepd. into the α-form, m. 163.5° (from EtOH), and the predominant β-form (XXXIII), m. 126° (from C₆H₆), by the virtual insolv. of the α-form in Et₂O. Attempted dehydration caused resin formation, esp. with XXXIII and 10% H₂SO₄, when a red resin was obtained. So hydrogenation gave 2,5-diphenyl-2,5-hexanediol as the α-form

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN (Continued)
 Pentamethylundecane. An attempted prep. by Grignard condensation of Me₂CHCH₂CHMeCH₂Br with EtOAc gave only secondary products, such as 2,4-dimethyl-6-hexanol, 2,4-dimethyl-1-pentene, and 2,4-dimethylpentane. (9) 2,4,6-Trimethyl-3,5-diisopropylheptane could not be prep. by Grignard condensation of (Me₂CH)₂CHBr (C.A. 41, 5850e) with EtOAc. (10) 2,7-Dimethyl-3,6-diisopropyldecane (X). (Me₂CH)₂CH₂O, formed by CrO₃ oxidn. of the alc., was condensed with VIII to 2,7-dimethyl-3,6-diisopropyl-4-octyne-3,6-diol, m. 109° (from CCl₄); the condensation of this ketone, doubly branched in the α-position, goes normally. Dehydration in 20% H₂SO₄ gave 2,7-dimethyl-3,6-diisopropyl-2,6-octadiene-4-yne (XI), b.p. 142°, d₂₀ 0.8360, n_D 1.5050, MR 77.34. Catalytic hydrogenation with Raney Ni at 180° and 120 kg./sq. cm. gave X, b.p. 142°, d₂₀ 0.7968, n_D 1.4470, mol. refraction 75.78; hydrogenation with Pt black gave instead 2,7-dimethyl-3,6-diisopropyl-4-octene (XII), b.p. 132°, d₂₀ 0.8157, n_D 1.4513, MR 73.99; hydrogenation with Raney Ni (room temp. and 1 atm.) gave intermediate products, 2,7-dimethyl-3,6-diisopropyl-2,4,6-octatriene, and 2,7-dimethyl-3,6-diisopropyl-3,5-octadiene, as well as XII. (11) 5,8-Dibutyldecane (XIII). BuMgBr and HCO₂Et gave 92% 5-nonanol, b.p. 103°, d₂₀ 0.8257, n_D 1.4289, MR 44.95, oxidized with CrO₃ to 5-nonanoic acid; condensation with VIII gave 83% 5,8-dibutyl-6-dodecenoic-5,8-diol, m. 130.5° (from EtOH). Dehydration in 25% H₂SO₄ gave 5,8-dibutyl-4,8-dodecadien-6-yne (XIV), b.p. 189°, d₂₀ 0.8222, n_D 1.4866, MR 95.77; total hydrogenation with Pt black in HOAc yielded XIII, b.p. 192°, d₂₀ 0.7922, n_D 1.4436, MR 94.48. Hydrogenation with Raney Ni in EtOH gave intermediate compds. (12) 2,9-Dimethyl-4,7-diisobutyldecane (XV), prep. similarly to XIII in 81% yield, b.p. 167°, d₂₀ 0.7882, n_D 1.4412, MR 94.52. 2,9-Dimethyl-4,7-diisobutyl-5-decyno-4,7-diol, m. 72° (from ligroin), was dehydrated in 25% H₂SO₄ to 2,9-dimethyl-4,7-diisobutyl-3,7-decadien-5-yne (XVI), b.p. 156°, d₂₀ 0.8023, n_D 1.4783, MR 95.94. Total hydrogenation to XV was possible only at a pressure of 130 kg./sq. cm. at 180° with Raney Ni. (13) The Wurtz reaction gave very poor yields of the desired hydrocarbons; the main products were dimers and "intermol. rearrangement" products of free radicals. Condensation of 1-bromodecane and iso-AmBr in Bu₂O with Na gave a small quantity of impure 2-methyltetradecane, b.p. 152°, d₂₀ 0.7832, n_D 1.4290, MR 71.75; the main products were Me₂CHCH₂ and the 2-methylbutenes, and the dimer of the decyl radical, eicosane. Instead of 2-methyl-4-isobutyldecane from 1-bromohexane and (Me₂CHCH₂)₂CHBr, only secondary products such as dodecane were formed. Tetradecane (XVII), and not the desired 2-methyl-4-isobutyldecane, was the main product from 1-bromohexane and (Me₂CHCH₂)₂CHBr, b.p. 135°, d₂₀ 0.7700, n_D 1.4303, MR 66.46. PhCH₂CH₂CHMe₂ and Na in xylene gave only "intermol. rearrangement" products and not the desired dimer, 2,7-dimethyl-4,6-dipropyldecane. Arylphenyl series: (1) 1,2-Diphenylheptane (XVIII). 1,2-Diphenylheptanol, from PhCH₂MgCl and BzH, m. 69° (from CCl₄); dehydration in 50% H₂SO₄ gave stilbene, m. 124° (from EtOH), and hydrogenation with Raney Ni easily formed XVIII, m. 52°. (2) 1,1-Diphenylpropane (XXIX). 1,1-Diphenyl-1-propanol (XX), in 87% yield from EtMgBr and PhBr, m. 96° (from 90% EtOH) [cf. Klages, Ber. 35, 2647 (1902)], was dehydrated by diatn. in vacuo with activated clay to 1,1-diphenyl-1-propene, b.p. 154°, d₂₀ 1.0250, n_D 1.5880, MR 63.70; hydrogenation with Raney Ni gave XIX, b.p. 145°, d₂₀ 0.9975, n_D 1.5701, MR 64.48. Dehydration of XX in boiling EtOH easily gave the dimer 2,3-dimethyl-1,1,4,4-tetraphenyl-1-butene, m. 212°; the structure was detd. by Br no., mol. wt. by f.p. in C₆H₆, and oxidn. (3) 1,2-Diphenylpropane (XXI). PhCH₂MgCl and AcPh gave 92%

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS ON STN (Continued)
 (XXXIV), m. 158.5°, and the β-form, m. 136°; dehydration of XXXIV in 20% H₂SO₄ gave (as with XXVII above) 2,5-diphenyl-2,5-epoxyhexane (2,5-dimethyl-2,5-diphenyltetrahydrofuran) (XXXV), b.p. 202°, d₂₀ 1.0380, n_D 1.5637, MR 73.29. The resistance of XXXV to catalytic hydrogenation was analogous to that of XXVIII; rupture of the heterocyclic ring with Na and EtOH gave 2,5-diphenyl-2-hexanol, b.p. 202°, d₂₀ 1.0125, n_D 1.5468, MR 74.52. Dehydration with 50% H₂SO₄ gave 2 olefins: a solid form, m. 139° (from EtOH), and a liq., b.p. 183°, d₂₀ 0.9916, n_D 1.5639, MR 77.62 (possible stereoisomers of 2,5-diphenyl-2-hexene) [cf. Klages, Ber. 35, 2633 (1902), and C.A. 1, 3005]. Attempts of hydrogenation of these olefins with Raney Ni did not form the desired XXXII, but instead cyclization gave trans-1,2-dimethyl-1,2-diphenylcyclobutane (XXXVI), b.p. 172°, m. 52.5°, and the cis-form (XXXVII), b.p. 172°, d₂₀ 0.9961, n_D 1.5540, MR 75.93; this reaction will be investigated further with larger amts. of starting material. (9) 1,1,2-Triphenylheptane (XXXVIII). Dehydration of Ph₂CH(OH)CH₂Ph (triphenylheptanol) (82% from PhBr and PhCH₂MgCl, m. 89°) with AcCl gave Ph₂CHCH₂Ph, triphenylheptylene, m. 73°, and hydrogenation with Na and AMOH then gave the desired XXXVIII, m. 54.5°. Cyclic series: (1) Decylcyclohexane (XXXIX). Catalytic hydrogenation of phenyldecane, 10% from 1-bromodecane, C₆H₆, and AlCl₃, b.p. 158°, d₂₀ 0.8978, n_D 1.5078, MR 72.36, with Pt black was strongly inhibited, apparently by traces of AlCl₃. BzH and C₉H₁₉MgBr gave 50% 1-phenyl-1-decanol, m. 34° (from EtOH), b.p. 189°, crystals greasy to the touch; equimol. amts. of PhCH₂OH and C₉H₁₈ were also formed. Dehydration with H₂SO₄ gave 1-phenyl-1-decene, b.p. 183-4°, d₂₀ 0.8726, n_D 1.4878, MR 71.29; hydrogenation of which then gave the desired XXXIX, b.p. 168°, d₂₀ 0.8167, n_D 1.4520, MR 73.98. The hydrogenation was selective, the intermediate phenyldecane being insol. in HOAc; the reaction then continued at 25% of the original rate. (2) 1,4-Diphenylcyclohexane could not be formed from C₅H₁₁Li and p-BrC₆H₄ with Na; only C₅H₁₁Li, C₅H₁₀, ClOH₂, and dimethylcyclohexane were formed. The condensation of C₅H₁₁MgBr with p-BrC₆H₄ with PhCl₃ (cf. C.A. 39, 2739, 6) was also unsuccessful. (3) 1,2-Dicyclohexylheptane (XL), obtained by the hydrogenation at 180° with Raney Ni of PhCH₂CH(OH)Ph, b.p. 140°, d₂₀ 0.8825, n_D 1.4802, MR 62.47. (4) 1,1-Dicyclohexylpropane (XLI), from PhCH₂CH₂CH₂OH by hydrogenation with Raney Ni, b.p. 141°, d₂₀ 0.9023, n_D 1.4935, MR 67.05. Condensation of EtCO₂Et and C₅H₁₁MgCl gave only secondary products: C₆H₁₀, C₆H₁₁CH₂Ac, and cyclohexylpropanol. (5) 1,2-Dicyclohexylpropane (XLII), from the hydrogenation of PhCH₂CHPhMe with Pt black, b.p. 148°, d₂₀ 0.8819, n_D 1.4791, MR 66.90. (6) 1,3-Dicyclohexylpropane (XLIII), from the hydrogenation of PhCH₂CH₂CHPh in HOAc with Pt black, b.p. 151°, d₂₀ 0.8740, n_D 1.4750, MR 67.01; the hydrogenation was nonselective, acting equally on the olefinic bond and the arocl. nuclei. (7) 1,2-Dicyclohexylbutane (XLIV), obtained from PhCH₂CH(OH)PhBr by hydrogenation with Raney Ni at 185°, b.p. 160°, d₂₀ 0.9146, n_D 1.5038, MR 71.85. (8) 1,4-Dicyclohexylbutane (XLV), obtained similarly from [CH₂CH(OH)Ph]₂ at 180°, b.p. 167°, d₂₀ 0.8731, n_D 1.4758, MR 71.69. (9) 1,2-Dicyclohexylpentane (XLVI), prep. similarly from PhCH₂CH(OH)PhBr, b.p. 157°, d₂₀ 0.9218, n_D 1.5092, MR 76.47. (10) 2,5-Dicyclohexylhexane (XLVII), from [CH₂CH(OH)PhMe]₂, b.p. 201°, d₂₀ 0.8901, n_D 1.4876, MR 80.87. (11) 1,1,2-Triphenylheptane was hydrogenated to 1,1,2-tricyclohexylheptane (XLVIII), b.p. 230°, d₂₀

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 1.0236, n20D 1.5786, MR 90.21. Studies of hydrocarbon properties as a function of mol. structure: Generally the aliph. hydrocarbons show increasing d. and n with increase in branching; the b. ps. decrease. The polyolefinic hydrocarbons show a trend in d. and n with the C/H ratio or degree of unsatn. The mol. exaltation due to the acetylenic group is roughly 3 times that for ethylenic bonds. The following phys. data (see also under each of the preceding individual syntheses), i.e., the mol. exaltation (obsd. mol. refraction - calcd. value), the kinematic viscosity in centistokes, and the abs. viscosity in centipoises, are given: XVII, -0.42, 3.45, 2.66; I, -0.01, 3.75, 2.35; V, -0.21, 4.33, 3.37; VI, -0.53, 3.71, 2.90; IX, -0.34, 4.65, 3.71; XI, -0.12, 10.28, 8.14; XII, -0.08, 9.64, 7.60; for the following only the mol. exaltation is given: IX, +4.04; XI, +4.19; XIV, +4.11; 5,8-dibutyl-4,6,8-dodecatriene, +2.02; 5,8-dibutyl-5,7-dodecadiene, +1.01; XVI, +4.28; 2,9-dimethyl-4,7-disobutyl-3,5,7-decatriene, +2.16; 2,9-dimethyl-4,7-disobutyl-4,6-decadiene, +1.02. The phys. state of the arylaliph. hydrocarbons is intimately bound to the mol. structure esp. the degree of symmetry.

Asym. compds. are liq. whereas the sym. ones are solid. The d. and n of liqs. decrease with increasing chain length (or decrease in C/H ratio) and with increasing degree of symmetry. Phys. data (read as above): XIX, +0.18, 5.07, 5.06; XXI, +0.12, 6.74, 6.61; XXII, +0.15, 7.81, 7.83; XXV, +0.06, 9.36, 9.15; XXIX, +0.20, 12.00, 11.58. With the cyclic hydrocarbons, the d. and n of the sym. isomers are lower than of the asym. ones. Phys. data (read as above): XXXIX, +0.05, 6.14, 5.01; XL, -0.01, 7.90, 6.96; XLI, -0.05, 9.68, 8.74; XLII, -0.20, 11.52, 10.16; XLIII, -0.09, 8.91, 7.80; XLIV, +0.13, 10.99, 10.05; XLV, -0.03, 12.71, 11.17; XLVI, +0.13, 14.68, 13.53; XLVII, -0.09, 23.35, 20.79; XLVIII, 0.00, 122.85, 127.80; XXXVI, 52.5°, b16 166°; XXXVII, b16 172°, -0.03, 41.56, 41.40. The viscosity of the 3 series above at 20° increases with the length of C chain, but not with the C/H ratio; the arylaliph. asym. hydrocarbons have lower values than the corresponding cyclic compds. An increase occurs with the appearance of closed rings, also with degree of symmetry for compds. of analogous structure. More evidence is needed to confirm these generalities. Conclusions: (1) The Wurtz reaction does not go so simply as given in the std. texts; free radicals play an important part. Temp. is important, esp. in the isomerization of the radicals, and it is difficult to isolate pure products, due to secondary products. (2) The Grignard condensation is a better method for obtaining the desired structures, with min. rearrangement of the reactants. (3) Dehydration and dehydrogenation depend greatly on mol. structure: the acetylenic diols dehydrate differently in the aliph. and arylaliph. series. After removal of the triple bond, epoxides are formed. Compds. of the type ArCH:CRAr are resistant to catalytic hydrogenation, so that selective redn. to arylaliph. or to cyclic hydrocarbons can be accomplished. Similarly, acetylenic diols are also selectively hydrogenated; the partial redn. of the triple bond is characterized by (1) a slower reaction, (2) change from exothermic to endothermic reaction, and (3) pptn. of the insol. olefin from EtOH. The hydrogenation of the sym. diaryl 1,4-epoxides is free of intramol. rearrangement.

ACCESSION NUMBER: 1948:27383 CAPLUS
 DOCUMENT NUMBER: 42:27383
 ORIGINAL REFERENCE NO.: 42:5833f-1,5834e-1,5835a-1,5836a-1,5837a-1,5838a-e
 TITLE: Aliphatic, arylaliphatic, and cyclic (C14-C20) hydrocarbons. Synthesis

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 AUTHOR(S): Tuot, Marcel; Guyard, Marcelle
 CORPORATE SOURCE: Ecole natl. superieure petrole, Strasbourg
 SOURCE: Bulletin de la Societe Chimique de France (1947)
 1086-96
 CODEN: BSCFAS; ISSN: 0037-8968
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

=> FIL STNGUIDE
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
57.80	58.01

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-7.80	-7.80

CA SUBSCRIBER PRICE

FILE 'STNGUIDE' ENTERED AT 11:35:30 ON 28 AUG 2007
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Aug 24, 2007 (20070824/UP).

=> d hist

(FILE 'HOME' ENTERED AT 11:23:08 ON 28 AUG 2007)

FILE 'CAPLUS' ENTERED AT 11:23:22 ON 28 AUG 2007

L1	4941 S OLEFIN AND DISTILLATION
L2	14686 S PROPANE AND (PROPENE OR PROPYLENE)
L3	207 S L1 AND PROPANE AND (PROPENE OR PROPYLENE)
L4	10 S L3 AND (EPOXIDATION OR EPOXIDE)

FILE 'STNGUIDE' ENTERED AT 11:35:30 ON 28 AUG 2007

=> s l3 and (compress or compressed)

	0 OLEFIN
	0 DISTILLATION
	0 PROPANE
	0 PROPENE
	0 PROPYLENE
	0 COMPRESS
	89 COMPRESSED
L5	0 L3 AND (COMPRESS OR COMPRESSED)

=> s l1 and (compress or compressed)

	0 OLEFIN
	0 DISTILLATION
	0 COMPRESS
	89 COMPRESSED
L6	0 L1 AND (COMPRESS OR COMPRESSED)

=> s l1

	0 OLEFIN
	0 DISTILLATION
L7	0 OLEFIN AND DISTILLATION

=> file caplus
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
1.44	59.45

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
0.00	-7.80

CA SUBSCRIBER PRICE

FILE 'CAPLUS' ENTERED AT 11:49:51 ON 28 AUG 2007

USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is held by the publishers listed in the PUBLISHER (PB) field (available for records published or updated in Chemical Abstracts after December 26, 1996), unless otherwise indicated in the original publications. The CA Lexicon is the copyrighted intellectual property of the American Chemical Society and is provided to assist you in searching databases on STN. Any dissemination, distribution, copying, or storing of this information, without the prior written consent of CAS, is strictly prohibited.

FILE COVERS 1907 - 28 Aug 2007 VOL 147 ISS 10
FILE LAST UPDATED: 27 Aug 2007 (20070827/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

<http://www.cas.org/infopolicy.html>

```
=> s l3 and (compress or compressed)
      3742 COMPRESS
      2128 COMPRESSES
      5734 COMPRESS
          (COMPRESS OR COMPRESSES)
      67481 COMPRESSED
L8      8 L3 AND (COMPRESS OR COMPRESSED)

=> d l8 1-8 abs ibib
```

L8 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

AB A low-pressure olefins recovery process and plant are described in which feed gas is compressed and distilled at a primary distillation pressure. The overhead stream is chilled at <30 kg/cm² to partially condense the overheads. The primary distillation tower is refluxed with at least a portion of the condensate. The overhead vapor is further chilled and partially condensed and the condensate is fed to a demethanizer. The remaining vapor is cooled in a cold section and the resultant liquid is phase separated and expanded to provide refrigeration for the cold section. The expanded vapor from the cold section is recycled to the process gas compressor. The bottoms streams from the primary distillation zone and the demethanizer are fractionated into resp. streams consisting essentially of ethylene, ethane, propylene, propane, C₄'s, and C₅. Process flow diagrams are presented.

ACCESSION NUMBER: 2006:8427 CAPLUS
DOCUMENT NUMBER: 144:88707
TITLE: Low-pressure olefin recovery process
INVENTOR(S): Verma, Vijender K.; Hu, Jichuan
PATENT ASSIGNEE(S): Kellogg Brown & Root, Inc., USA
SOURCE: U.S. Pat. Appl. Publ., 19 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2006004242	A1	20060105	US 2004-884659	20040702
CA 2497943	A1	20060102	CA 2005-2497943	20050222
IN 2005K000152	A	20070202	IN 2005-K0152	20050310
EP 1637577	A2	20060322	EP 2005-5372	20050311
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, BA, HR, IS, YU				
CN 1715260	A	20060104	CN 2005-10082196	20050701
MX 2005PA07272	A	20060111	MX 2005-PA7272	20050701
PRIORITY APPLN. INFO.:			US 2004-884659	A 20040702

L8 ANSWER 3 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

AB The process of U.S. 2,600,110 (CA 46, 8358n), which is based on the heat-pump principle, is improved by passing a major portion of the compressed vapors of the kettle product refrigerant to the kettle section of the fractionator and compressing the rest of the vapors in a 2nd compression step so that they are condensed by available cooling water, thus increasing the efficiency of heat transfer. The improved process is particularly adapted to the separation of C₂H₄ from C₂H₆, C₃H₆ from C₃H₈, and C₄H₈ from C₄H₁₀.

ACCESSION NUMBER: 1966:57911 CAPLUS
DOCUMENT NUMBER: 64:57911
ORIGINAL REFERENCE NO.: 64:10793d-e
TITLE: Fractional distillation
INVENTOR(S): Palen, Joseph W.; Moon, John J.
PATENT ASSIGNEE(S): Phillips Petroleum Co.
SOURCE: 5 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3229471		19660118	US 1961-160066	19611218
PRIORITY APPLN. INFO.:			US	19611218

L8 ANSWER 2 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

AB Ethylene (I) and propylene (II) are separated from the raw gas, formed from pyrolysis of hydrocarbons, by washing the raw gas at 8-12 atmospheric with a N-alkyl-γ- or δ-lactam to dissolve acetylenes, diolefins, H₂S, S-containing organic compds, and C₄ hydrocarbons. The washed gas contains I, II, H₂S, CO, H, CH₄, CO₂, ethane, propane, and butane. Preferred lactam for washing is N-methylpyrrolidinone (III). The washed gas is treated with an aqueous alkali solution to remove traces of H₂S and CO₂. The purified gas is compressed to 25-35 atmospheric, and either condensed by cooling it at very low temperature, or absorbed by a liquid. The condensate or the solution is distilled to give pure I and II. The components absorbed by III, namely, acetylene, diolefins, H₂S, S-containing organic compds, and a part of CO₂, are regenerated by evaporation. The regenerating column is a continuous distillation column, fed with the washing liquor and with steam at such a rate that at the head of the column a part of water and the regenerated components are distilled. The III in the residue contains 1-10% water. The hot residual III is dehydrated by bubbling through it a part of CH₄ and of H, released in the purification. Acetylene and diolefins are partly polymerized during the regeneration. The consumption of alkali is low. A design of the plant is given.

ACCESSION NUMBER: 1966:419864 CAPLUS
DOCUMENT NUMBER: 65:19864
ORIGINAL REFERENCE NO.: 65:3649f-h
TITLE: Separation of ethylene and propylene from raw pyrolyzed gas
INVENTOR(S): Thormann, Kurt
PATENT ASSIGNEE(S): Metallgesellschaft A.-G.
SOURCE: 5 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1183902		19641223	DE 1960-M45718	19600623
PRIORITY APPLN. INFO.:			DE	19600623

L8 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

AB Oxidation products, e.g. ethylene oxide, acrolein, and methacrolein, are prepared by the controlled catalytic oxidation of corresponding olefinic hydrocarbons with O₂. For example, the reaction mixture obtained by the catalytic oxidation of propylene with O₂ in the vapor phase in the presence of a Cu₂O catalyst and quenching with H₂O contains essentially 2.6% acrolein, 34.2% propylene and propane, 0.8% O₂, 19.4% permanent gases, and 43% water. The mixture is cooled from 149° to 49° in 2 stages. Two separate liquid condensates are obtained, the first containing 8.8% acrolein and 76% water and the second containing 4.2% acrolein and 13% water. The remaining vapor phase contains 87% acrolein, 1.3% water, the hydrocarbons that are gaseous under normal conditions, and the permanent gases of the reaction mixture. The vapor phase is compressed from its pressure of 5.6 atmospheric to 21 atmospheric and is introduced into an absorption tower at the bottom. A jet of water is introduced at the top. The first liquid condensate, separated during the quenching of the reaction mixture, is introduced into the absorption tower at a place midway between the water inlet and the vapor phase inlet. The absorption tower is maintained at 21° and 21 atmospheric. Acrolein is selectively absorbed by the aqueous solvent, forming a solution of water and acrolein in a proportion of 50.1 moles/water/mole acrolein. The gases leaving at the top of the tower contain essentially propylene, propane, and permanent gases. The absorption solution is withdrawn from the bottom of the tower and is distilled with the second condensate. The top fraction from the distillation contains acrolein and a relatively small amount of water. More than 99% acrolein is recovered from the reaction mixture. The aqueous phase at the bottom of the column is partially returned to the absorption tower. A total of 17.6 moles water are introduced at the top of the tower per mole of acrolein which is recovered by distillation. If this reaction is conducted under essentially the same conditions, with the exception that the vapor phase is introduced into the absorption tower without previous compression and both liquid condensates are distilled directly in the distillation column, 35.2 moles water are required per mole acrolein to obtain an equivalent yield of acrolein.

ACCESSION NUMBER: 1964:16023 CAPLUS
DOCUMENT NUMBER: 60:16023
ORIGINAL REFERENCE NO.: 60:2772a-e
TITLE: Recovery of water-soluble oxidation products of hydrocarbons
INVENTOR(S): Courter, Martin L.; Thayer, David S.
PATENT ASSIGNEE(S): Shell Internationale Research Maatschappij N. V.
SOURCE: 6 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1147932		19630502	DE 1961-873353	19610406
US 3097215		19630709	US 1960-21003	19600408

LB ANSWER 4 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
AB A normally gaseous hydrocarbon component is separated from a mixture of normally gaseous hydrocarbons by introducing the mixture into a distillation zone. A bottom fraction and a vaporous overhead fraction are withdrawn from the distillation zone. A portion of the overhead fraction, which comprises the component which is to be separated, is compressed and thereby heated. A portion of the compressed overhead fraction, which has been further heated or cooled, is brought into contact indirectly with liquid from the lower portion of the distillation zone thereby heating and partially vaporizing the liquid from the distillation zone and partially condensing the overhead. The condensed overhead may be used to reflux the distillation zone, while the vaporized liquid may be used to reboil the distillation zone. This process may be used in the separation of C2H4 from C2H6 in a C2-splitting operation or for the separation of C3H6 from C3H8.

ACCESSION NUMBER: 1962:455562 CAPLUS
DOCUMENT NUMBER: 57:55562
ORIGINAL REFERENCE NO.: 57:10979a-c
TITLE: Gas separation, especially in production of ethylene and propylene
INVENTOR(S): Greco, Saverio G.
PATENT ASSIGNEE(S): M. W. Kellogg Co.
SOURCE: 6 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3000189	---	19610919	US 1956-622291	19561115

PRIORITY APPLN. INFO.: US 19561115

LB ANSWER 5 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB Chilling of alkylation mixts. rich in isobutane is accomplished by flash vaporization or "self refrigeration." Vapors rich in isobutane are then compressed and fed to high-pressure fractionating towers and isobutanerich liquids returned to the reactors. The temperature of the system under the proposed conditions is maintained lower than usual and decomposition of acidic components in the alkylate is reduced during deisobutanization. The isobutane and olefinic material are treated with a catalyst in the liquid phase at <100°F. With a H2SO4 catalyst, alkylation temps. of 30-55°F. are preferred. The alkylatable material is usually an olefinic hydrocarbon, e.g. propylene or butylene, or it may be an alkyl ester. For example, 30.2 bbl. propylene, 19.2 bbl. butylene, and 539.8 bbl. isobutane were treated in the liquid phase at 50 lb./in.2 gage with 92% H2SO4. The reaction mixture was cooled by indirect heat-exchange with flashed effluent hydrocarbons, separated, and deisobutanized.

ACCESSION NUMBER: 1963:440793 CAPLUS
DOCUMENT NUMBER: 59:40793
ORIGINAL REFERENCE NO.: 59:7292f-g
TITLE: Alkylation of olefinic hydrocarbons
INVENTOR(S): Davies, James A.
PATENT ASSIGNEE(S): Texaco Inc.
SOURCE: 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3097250	---	19630709	US 1960-39370	19600628

PRIORITY APPLN. INFO.: US 19600628

LB ANSWER 7 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB Isobutane (60-90 volume % of the hydrocarbon charge) and olefins, such as propene, react in the liquid phase in the presence of a nonvolatile alkylation catalyst, e.g. H2SO4 (88-94 weight % titratable acidity). The temperature and pressure conditions (30-75°F. and 0-30 lb./sq. in. gage) are such that part of the isobutane and practically all of the C3H8 and lighter products are evaporated to give effective refrigeration for the alkylation zone. The alkylator delivers a depropanized liquid emulsion to be settled and caustic-washed free of acid. This alkylate passes to the top of a distillation tower where butanes are distilled from the downcoming alkylate and returned to the alkylator. The isobutane-rich vapors from the alkylator containing C3H8 are compressed (40-130 lb./sq. in. gage) and condensed with cooling water. The condensate is depropanized, and the isobutane-rich bottoms are returned to the alkylator.

ACCESSION NUMBER: 1962:443938 CAPLUS
DOCUMENT NUMBER: 57:43938
ORIGINAL REFERENCE NO.: 57:8811f-h
TITLE: Catalytic alkylation of olefins with isobutane
PATENT ASSIGNEE(S): Texaco Development Corp.
SOURCE: 9 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 895178	---	19620502	GB 1960-15908	19600505

PRIORITY APPLN. INFO.: US 19590511

LS ANSWER 8 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

AB The liquid mixture of hydrocarbons containing olefins, e. g., propane and propylene, is freed of these components by successive decantations in a specially designed apparatus. There the olefinic components and the esterifying solution (a 75% H₂SO₄ solution) are allowed to circulate in countercurrent manner by means of flowing in an inert gas preliminarily compressed. The temperature in the battery is 40° and the pressure 20 kg. per sq. cm. At one end of the battery is collected a liquid containing saturated hydrocarbons and practically free of olefins, and at the other an esterifying solution saturated with olefin alc. which is caused to appear there by dilution and heating. The liberated alcs. are isolated by suitable means, e. g., distn. or solvent extraction. Drawing of apparatus is included.

ACCESSION NUMBER: 1944:25045 CAPLUS
DOCUMENT NUMBER: 38:25045
ORIGINAL REFERENCE NO.: 38:36641,3665a-b
TITLE: Esterification of olefins
INVENTOR(S): Guinot, Henri M.
PATENT ASSIGNEE(S): Alien Property Custodian.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2345114		19440328	US	

=> FIL STNGUIDE
COST IN U.S. DOLLARS

SINCE FILE ENTRY	TOTAL SESSION
28.07	87.52

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE ENTRY	TOTAL SESSION
-6.24	-14.04

CA SUBSCRIBER PRICE

FILE 'STNGUIDE' ENTERED AT 11:51:50 ON 28 AUG 2007
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Aug 24, 2007 (20070824/UP).

=> log hold
COST IN U.S. DOLLARS

SINCE FILE ENTRY	TOTAL SESSION
1.20	88.72

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE ENTRY	TOTAL SESSION
0.00	-14.04

CA SUBSCRIBER PRICE

SESSION WILL BE HELD FOR 120 MINUTES
STN INTERNATIONAL SESSION SUSPENDED AT 12:04:03 ON 28 AUG 2007